

Separation of Bitumen from Utah Tar Sands by a Hot Water Digestion-Flotation Technique

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Tar sand deposits in the state of Utah contain more than 25 billion bbl of in-place bitumen. Although 30 times smaller than the well-known Athabasca tar sands, Utah tar sands do represent a significant domestic energy resource comparable to the national crude oil reserves (31.3 billion bbl). Based upon a detailed analysis of the physical and chemical properties of both the bitumen and the sand, a hot-water separation process for Utah tar sands is currently being developed in our laboratories at the University of Utah. This process involves intense agitation of the tar sand in a hot caustic solution and subsequent separation of the bitumen by a modified froth flotation technique. Experimental results with an Asphalt Ridge, Utah, tar sand sample indicated that percent solids and caustic concentration were the two most important variables controlling the performance of the digestion stage. These variables were identified by means of an experimental factorial design, in which coefficients of separation greater than 0.90 were realized. Although preliminary in nature, the experimental evidence gathered in this investigation seems to indicate that a hot-water separation process for Utah tar sands would allow for the efficient utilization of this important energy resource.

The projected increase in the ever-widening gap between the domestic energy demand and the domestic energy supply for the next few years has motivated renewed interest in energy sources other than petroleum, such as tar sands, oil shale and coal. Although a number of research programs on the exploitation of national coal and oil shale resources have already been completed, very few programs have been initiated on the processing of tar sand resources in the United States. In recognition of their significance as a domestic energy resource, investigators at the University of Utah have designed an extensive research program on Utah tar sands. An important phase of this program, and the main subject of this publication, is the development of a hot-water process for the recovery of bitumen from Utah tar sands, as a preliminary step toward the production of synthetic fuels and petrochemicals.

The term "tar sand" refers to a consolidated mixture of bitumen (tar) and sand. The sand in tar sand is mostly α -quartz as determined from X-ray diffraction patterns. Alternate names for "tar sands" are "oil sands" and "bituminous sands." The latter is technically correct and in that sense provides an adequate description.

Tar sand deposits occur throughout the world, often in the same geographical areas as petroleum deposits. Significantly large tar sand deposits have been identified and mapped in Canada, Venezuela and, the United States.¹

By far, the largest deposit is the Athabasca tar sands in the Province of Alberta, Canada. According to the Alberta Energy Resources Conservation Board (AERCB),^{2,3} proved reserves of

crude in-place bitumen in the Athabasca region amount to almost 900 billion bbl. To date, this is the only tar sand deposit in the world being mined and processed for the recovery of petroleum products. Great Canadian Oil Sands, Ltd. (GCOS) produces 20 million bbl of synthetic crude oil per year. Another plant being constructed by Syncrude Canada, Ltd. is expected to produce in excess of 40 million bbl of synthetic crude oil per year.

According to the Utah Geological and Mineral Survey (UGMS), tar sand deposits in the state of Utah contain more than 25 billion bbl of bitumen in place, which represent almost 95% of the total mapped resources in the United States.⁴ The extent of Utah tar sand reserves seems small compared to the enormous potential of Canadian tar sands. Nevertheless, Utah tar sand reserves do represent a significant energy resource comparable to the United States crude oil proved reserves of 31.3 billion bbl in 1976.⁵

Tar sands in Utah occur in 51 deposits along the eastern side of the state.⁴ However, only six out of these 51 deposits are worthy of any practical consideration (Fig. 1). As indicated in Table 1, Tar Sand Triangle is the largest deposit in the state and contains about half of the total mapped resources.

Information regarding the grade or bitumen content of Utah deposits is still very limited. The bitumen content varies significantly from deposit to deposit, as well as within a given deposit. In any event, the information available⁶⁻⁸ seems to indicate that Utah deposits are not as rich in bitumen as the vast Canadian deposits which average 12 to 13% by weight.⁹ Although many occurrences of bitumen saturation up to 17% by weight have been detected in the northeastern part of the state (Asphalt Ridge and P. R. Spring), the average for reserves in Utah may well be less than 10% by weight.

Separation Technology

As in any other mining problem, there are two basic approaches to the recovery of bitumen from tar sands. In one

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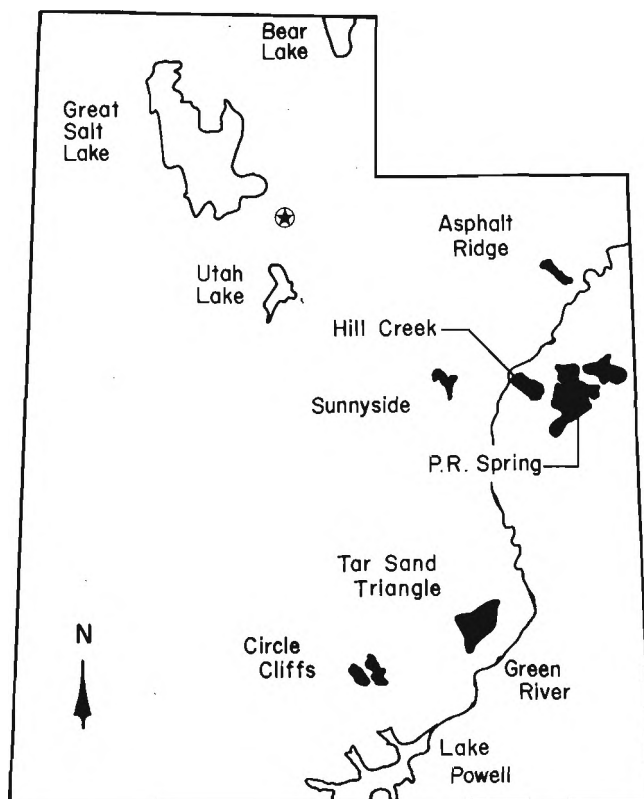


Fig. 1. Major tar sand deposits in the state of Utah.

case, the ore can be mined and transported to a processing plant where the bitumen is recovered. Alternately, the bitumen can be processed "in-situ." Over the last 50 years, a great variety of separation processes, including both surface mining and in-situ processes, have been proposed in the literature.⁹⁻¹¹ However, until recent years, little was known about the potential applicability of any of these processing strategies to Utah tar sands.

Based on the fact that the hot-water process is the only process whose technology is well developed and is currently being used in the primary separation of bitumen from Canadian tar sands; the main objective in this investigation was to quantify the performance of a similar process for Utah tar sands. In particular, research efforts were concentrated on the identification of the main operating variables which control the quality of the separation achieved by hot-water processing.

The hot-water process was first described by K. A. Clark in 1923¹² and has been repeatedly modified thereafter.¹³⁻¹⁵ As applied to Utah tar sands, this process basically consists of the separation and recovery of bitumen from tar sands by digesting the raw material with a hot aqueous solution containing a caustic wetting agent such as sodium hydroxide, sodium carbonate, or sodium silicate. The resulting strong surface hydration and shearing forces operative at the sand-bitumen interface give rise to the displacement and subsequent disengagement of the bitumen by the aqueous phase (Fig. 2). Once the bitumen has been displaced and the sand particles have been liberated, the two phases can be separated by a modified froth flotation technique based on the natural hydrophobicity exhibited by the free bituminous droplets. The name of the process arises from the fact that digestion is accomplished at temperatures just below the boiling point of water.

In practice, the application of the hot-water processing technique to Utah tar sands results in a two-stage process (Fig. 3). In the first stage, referred to as digestion, crushed tar sand is

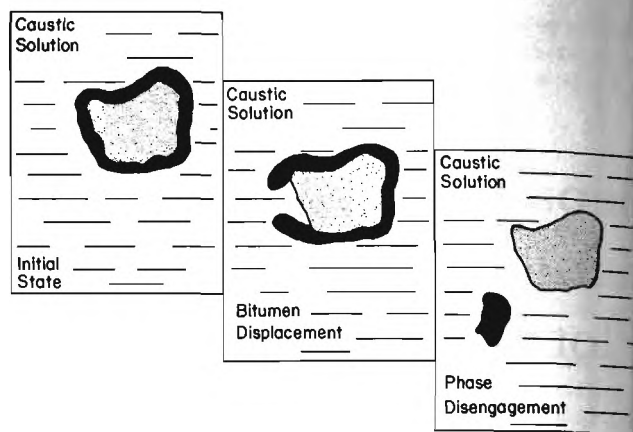


Fig. 2. The hot-water process: schematic representation of bitumen displacement and phase disengagement during digestion.

contacted with the hot caustic solution in a stirred tank reactor, at constant temperature, for a specified digestion time. Because of the highly viscous nature of the bitumen in Utah tar sands, the impeller in the reactor must be designed so as to produce a high-shear force field capable of rupturing the bituminous film coating the sand particles (Fig. 2), so that the solid phase may become exposed to the caustic solution. Upon contact, the caustic solution wets the surface of the sand particles and the system reaches its desired equilibrium configuration, i.e., the solid and bituminous phases being separated by the aqueous phase. Ideally, at the end of the digestion stage, the bituminous phase not only has been displaced but also completely disengaged from the solid phase, as a result of the strong shearing action by the impeller in the digester.

The second stage, flotation, is based on the hydrophobic nature of the bitumen at moderate pH values. In the flotation cell, air bubbles attach to the free bituminous droplets and carry them to the top of the cell, while the hydrophilic sand particles settle to the bottom. The flotation cell is operated at relatively low stirring speed so as to reduce the contamination of the bitumen concentrate with very fine sand particles. In actual processing, the hydrophobic bitumen concentrate removed from the top of the flotation cell would be sent to a refining plant for upgrading. On occasions, relatively large lumps of nonfloatable, undigested tar sand may be found with the sand tailings. This material can be recovered from the tailings simply by screening. The scavenger concentrate so produced has a grade sufficiently high to either be recycled or refined as is.

The mechanism or means by which the bitumen is displaced from the surface of the sand particles is not yet well understood and, as a result, a useful theoretical framework does not exist. Furthermore, although similar in principle, the actual separation mechanisms in the processing of Athabasca and Utah tar sands appear to differ significantly. Because of the high moisture content of Athabasca tar sands, 3-5% by weight connate water, it has been postulated by Canadian investigators that the equilibrium structure of Athabasca tar sands consists of sand particles separated from the bitumen by a film of connate water surrounding the sand particles.^{3,9,10} Accordingly, the bitumen in Athabasca tar sands has already been displaced from the sand by the connate water. Under these conditions, bitumen separation reduces to a relatively simple phase disengagement process. Unlike Athabasca tar sands, Utah tar sands are so dry that their moisture content cannot be detected by standard analytical techniques. Obviously, in the absence of water, the bitumen is directly in contact with, and bonded to, the surface of the sand particles. Hence, hot-water processing of

Table 1. Extent of Utah Tar Sand Deposits.

Deposit	Location	In-Place Bitumen ⁽⁴⁾ (billion bbl)
Tar Sand Triangle	SE, Utah	12.5 - 16.0
V. R. Spring	NE, Utah	4.0 - 4.5
Unayside	NE, Utah	3.5 - 4.0
Circle Cliffs	SE, Utah	1.3
Hill Creek	NE, Utah	1.2
Asphalt Ridge	NE, Utah	1.0

Utah tar sands involves both bitumen displacement and phase disengagement phenomena (Fig. 2).

Several investigators^{16,17} have attempted to explain bitumen displacement based on a surface energy balance postulating that, in order for the bitumen to be displaced, the total free energy of the system must decrease. However, this thermodynamic approach attempts to describe only the final equilibrium state and, in doing so, it fails to account for possible physical and electrostatic kinetic barriers which seem to be operative in the system.^{16,17} Other investigators¹⁸⁻²¹ have analyzed the displacement phenomenon by consideration of a chemical reaction between the hydrated silica surface and the hydroxy radicals present in the caustic solution, which in turn results in the displacement of the bitumen from the sand particles. Although this second approach eventually could provide a more realistic description of the displacement mechanism and rate phenomenon, it is found to be very limited because of the complexity of the interactions between the different phases of the sand-bitumen-water system. As a result, the process designer is left with one alternative, namely, the development of empirical models based on laboratory and/or pilot plant extraction tests. This is, in essence, the method of approach adopted in this investigation.

Definition of the Research Problem

Ideally, the development of a new processing strategy should naturally arise from a thorough understanding of its fundamentals. However, as emphasized previously, a useful theoretical model capable of describing the different phenomena occurring during hot-water processing of tar sands does not exist.

In view of the foregoing consideration, the best alternate approach would be the direct application of the well-developed Canadian technology to Utah tar sands. However, because of significant differences in the physical and chemical nature of Canadian tar sands as compared to Utah tar sands, and because of considerable differences in climatic conditions between the two locations, the separation technology to be developed for the processing of Utah tar sands and the technology already being used for the processing of Canadian tar sands are expected to be substantially different. Under these circumstances, an independent investigation on the processing of Utah tar sands, based on laboratory and/or pilot plant separation tests, was considered to be a preferable approach.

Owing to the large number of variables controlling the performance of the hot-water process, the effect of each individual variable on the overall performance could not be studied separately, as this would involve considerable experimental effort. Instead, experimental design techniques in which the number of experiments is reduced to a minimum were used, in combination with the Box and Wilson optimization algorithm.²²⁻²⁵ The Box and Wilson algorithm for experimental optimization has been successfully applied in a wide variety of industrial research problems.²⁶⁻²⁸ In this

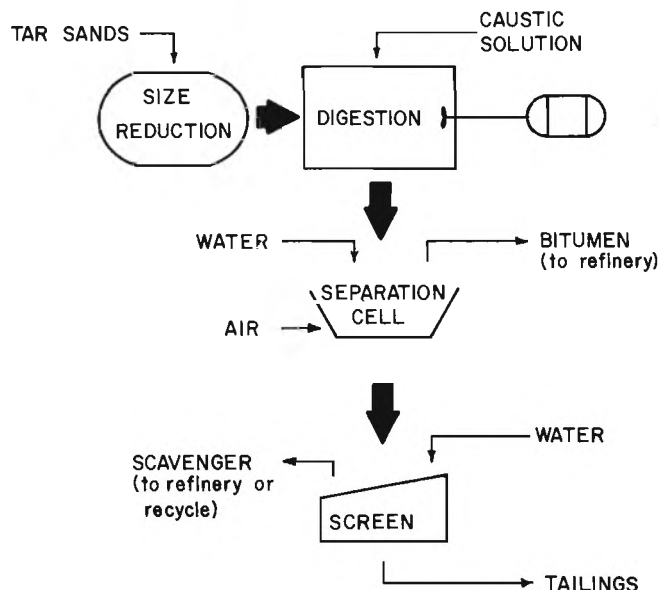


Fig. 3. Modified hot-water process for the separation of bitumen from Utah tar sands.

particular application, the primary objectives were to determine the optimum experimental conditions for the separation of the bitumen from the sand and to identify the main digestion variables controlling the quality of this separation. A detailed description of the Box and Wilson algorithm and its application to Utah tar sands may be found elsewhere.²⁹

Operation Variables Controlling the Performance of the Hot-Water Process: The number of variables affecting the performance of each of the two stages of the hot-water process (digestion and flotation) is relatively large. A listing of these variables is presented in Table 2.

In this particular study, flotation conditions were kept constant in all experiments, so that the effect of digestion variables on the overall system response could be studied first. With this experimental approach, flotation, while being an integral part of the process, was also used as an analytical tool for the evaluation of the effectiveness of the digestion stage.

Similarly, discrete digestion variables (feed source, wetting agent, and reactor design) were not varied during the experimental optimization process. However, once the optimum conditions were determined for an Asphalt Ridge sample, the effect of other feed sources on the quality of the separation was studied in a separate series of experiments.

As a first approximation to the processing of Utah tar sands, this investigation was mainly concerned with the equilibrium response of the system. Under these circumstances, dynamic variables such as digestion time and feed size did not deserve primary consideration, since they would not influence the quality of the separation at equilibrium.

Finally, based on the fact that bitumen viscosity decreases significantly with temperature (see Fig. 9), the temperature in the digester was kept constant at 95°C, just below the boiling point of water. The effect of lower temperatures on the quality of the separation was also studied in a separate series of experiments.

In summary, of all the operation variables listed in Table 2, only three are left for consideration, namely, the percent solids in the digester, the concentration of caustic in solution, and the intensity of agitation (stirring speed). In this regard, the term "percent solids" refers to the amount of tar sand (both bitumen and sand) with respect to the total mass of material in the system.

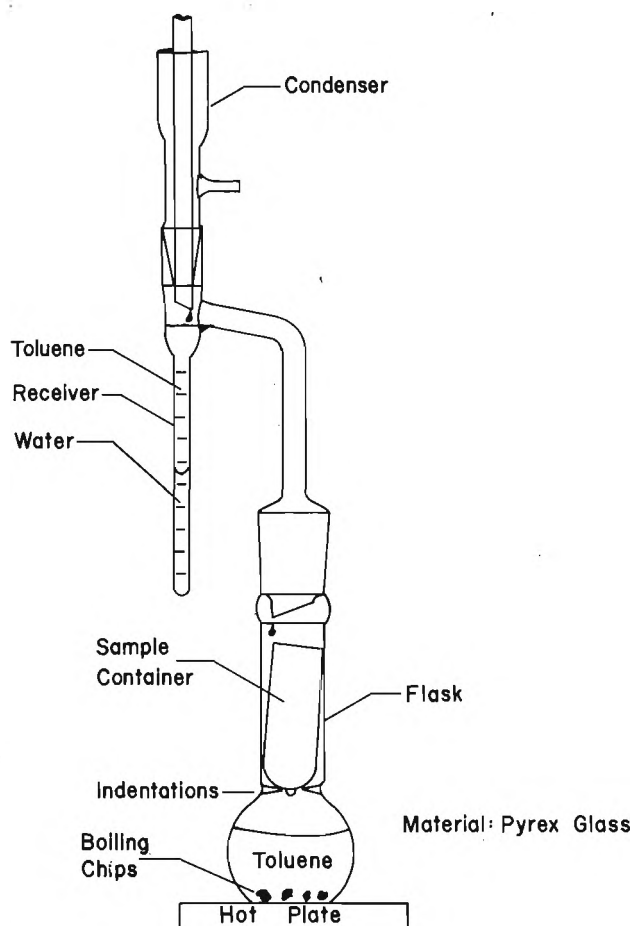


Fig. 4. Dean and Stark tube assembly.

Specification of an Objective Function for Optimization: The actual implementation of any optimization algorithm generally entails the representation of the economical or technical objective (maximize profit, minimize cost, maximize efficiency, etc.) in terms of a set of mathematical quantities. The resulting relationship is normally referred to as the objective function. In this particular application, the primary optimization objective was to maximize the quality of the separation achieved by hot-water processing of Utah tar sands.

Since the hot-water process is, in essence, a separation process, separation indices used to quantify the quality of the separation in other similar processes can also serve as a technical objective function for the system under consideration. In 1970, Schulz³⁰ published an excellent review of the most frequently used separation indices in the mineral industry. Because of its clear physical significance, the coefficient of separation (defined as the fraction of the feed material which undergoes a perfect separation while the rest of the feed is distributed unchanged into the respective product streams³⁰) provided an adequate, "one-parameter" description of the overall process performance and was selected as the objective function for process optimization.

The coefficient of separation can be simply evaluated as the difference between the recovery of the valuable component (bitumen) in the concentrate and the recovery of gangue (sand) in the same concentrate.

Experimental

The processing strategy used in laboratory hot-water separation tests is schematically represented in Fig. 3. Before

Table 2. Operation Variables Controlling the Performance of the Hot-Water Process

Digestion:	Discrete:	D1. Feed Source
		D2. Wetting Agents
		D3. Impeller and Reactor Design
	Continuous:	V1. Temperature
		V2. Percent Solids
		V3. Digestion Time
Flotation:	Discrete:	V4. Wetting Agent Addition
		V5. Feed Size
		V6. Intensity of Agitation
	Continuous:	E1. Cell Design
		E2. Flotation Reagents
		W1. Percent Solids
		W2. Intensity of Agitation
		W3. Temperature
		W4. Solution pH
		W5. Flotation Reagents Additions
		W6. Air Flowrate

Table 3. Average Bitumen Content of Utah Tar Sand Samples

Deposit	Average Bitumen Content (%, by weight)
Asphalt Ridge	13.1
P. R. Spring	12.2
Sunnyside	9.0
Tar Sand Triangle	5.0

each series of experiments was initiated, the tar sand samples were thoroughly mixed in large batches in order to homogenize the quality of the feed. A fraction of this material was then extruded down to smaller pieces ($\sim 3/8$ -in.) and fed to a 1-gal, stirred tank reactor where it was contacted with the hot caustic solution and stirred, at constant temperature, for a specified digestion time. The essential features of this reactor are an impeller with two opposing pitched blade turbines (4-in. OD), a torque meter, an SCR speed controller, a tachometer, a reflux-takeoff condenser, and a temperature control/heating system. The amount of tar sand and solution added to the digester in each separation test was calculated so that the active volume of the reactor was close to a gallon in all of the experiments. At the end of the digestion stage, the bitumen had been displaced from the surface of the sand particles and could be separated from the dispersed sand in a conventional batch bench-scale flotation machine where the bitumen was floated with air.

In the flotation cell, the percent solids was reduced to about 20%, by weight, by the addition of an aqueous caustic solution at room temperature ($\sim 15^\circ\text{C}$). The alkalinity of the solution was adjusted in order to maintain the pH of the suspension above 10. This relatively high flotation pH allowed for the flocculation of the sand tails and a faster flotation response. Besides sodium hydroxide, no other reagent was added to the flotation cell. The intensity of agitation of the suspension was maintained at a relatively low level, compared to conventional mineral flotation practice, in order to avoid contamination of the concentrate with fine sand particles. The hydrophobic bitumen concentrate was removed from the top of the flotation cell while the hydrophilic free sand particles were recovered from the bottom of the cell.

Representative samples of the feed, concentrate, and tailings were analyzed to determine their composition with respect to bitumen, sand, and water. For this purpose, several Dean and Stark tube assemblies (Fig. 4) were set up in accordance with the procedure reported by the US Bureau of Mines.³¹ A weighed

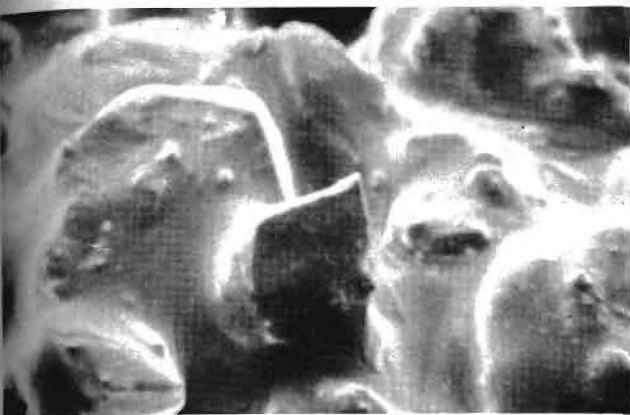


Fig. 5. Scanning electron micrograph of an Athabasca, Canada, tar sand sample. Bitumen content: 16% by weight. ~ 400X.

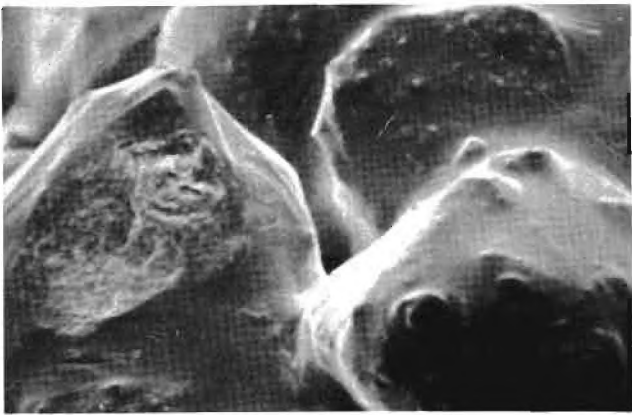


Fig. 6. Scanning electron micrograph of an Asphalt Ridge, Utah, tar sand sample. Bitumen content: 13% by weight. ~ 400X.

sample, contained in a double thickness, cellulose extraction thimble, was placed in the neck of a specially designed receiver flask held by four indentations. About 200 ml of reagent grade toluene were added to the flask and heated to boiling. Toluene vapors dissolved the bituminous materials in the sample and also removed any trace of water present. The vapors were trapped by the condenser and returned to the system. Because of the immiscibility and different density of the two phases in the toluene-water system, water droplets settled through the condensate and were collected in the capillary tube, while the toluene was refluxed. After 4 to 6 hr, extraction reached completion and the volume of water in the sample was read on the graduated capillary. The cellulose thimble was then dried and weighed to determine the amount of solids left in it and the bitumen content was calculated by difference. The analytical technique proved to have very good reproducibility, the coefficient of variation being less than 1%.

Experimental Results and Discussion

Preliminary separation tests indicated that, under similar operating conditions, the quality of the separation was strongly dependent upon the nature of the tar sand. In fact, while excellent results, comparable to those obtained in the processing of Athabasca tar sands, were obtained with two high-grade Utah samples (Asphalt Ridge and P.R. Spring), hot-water separation tests of low-grade tar sands (Sunnyside and Tar Sand Triangle) were not at all successful. Accordingly, tar sand samples from these four Utah deposits were characterized in terms of their bitumen content, bitumen viscosity, and particle size distribution of the sand.

Average bitumen content of samples from four different Utah tar sand deposits are presented in Table 3. Although these samples may not be representative of the deposit as a whole, the grades reported in Table 3 are in good agreement with previous results which show that northeastern Utah deposits, and especially the Uintah Basin deposits (Asphalt Ridge and P.R. Spring) have higher bitumen content than Tar Sand Triangle in southeastern Utah.⁷

Scanning electron micrographs of tar sand samples from Athabasca, Canada, and three northeastern Utah deposits are presented in Fig. 5-8. As compared to the Sunnyside sample (Fig. 8), the bituminous film in the Athabasca, Asphalt Ridge, and P.R. Spring samples (Figs. 5-7, respectively) appears to be much thicker and continuous throughout the sample.

The viscosity of the bitumen in tar sands is of primary importance to the design of in-situ mining, recovery, upgrading, and material handling operations. Experimentally determined flow curves of Asphalt Ridge and Athabasca



Fig. 7. Scanning electron micrograph of a P.R. Spring, Utah, tar sand sample. Bitumen content: 12% by weight. ~ 400X.

bitumen, at various temperatures, demonstrated the Newtonian nature of both fluids.²⁹ Perhaps of more practical significance is the fact that the viscosity of the Utah (Asphalt Ridge) bitumen is about two orders of magnitude greater than the viscosity of the Canadian (Athabasca) bitumen in the temperature range studied, as illustrated in Fig. 9. This accounts for the fact that Athabasca tar sands can be digested in a conventional rotary drum, while Utah tar sands seem to require intense shear conditions.

Viscosity measurements of Athabasca bitumen at different temperatures are in good agreement with data previously reported in the literature.^{9,31,32} An apparent activation energy on the order of 28 kcal/mole was calculated from the data in Fig. 9, indicative of the fact that momentum transfer is accompanied by rather significant structural transformations, as temperature increases.

The particle size distribution of the solids in Utah tar sands was determined by conventional sieving techniques in the size range of 590 μm down to 37 μm . Particle size distributions of the sand in several Utah tar sand deposits are compared in Fig. 10. According to the Canadian size classification,^{9,32} the sands in Utah tar sand deposits are comparable in size to the Class II Athabasca sands which are the richest tar sands in the McMurray formation.³²

Preliminary Experimental Results: Preliminary results indicated that effective displacement and disengagement of bitumen from both Asphalt Ridge and P.R. Spring tar sand samples could be achieved by hot-water processing. The overall composition of the concentrate removed from the separation

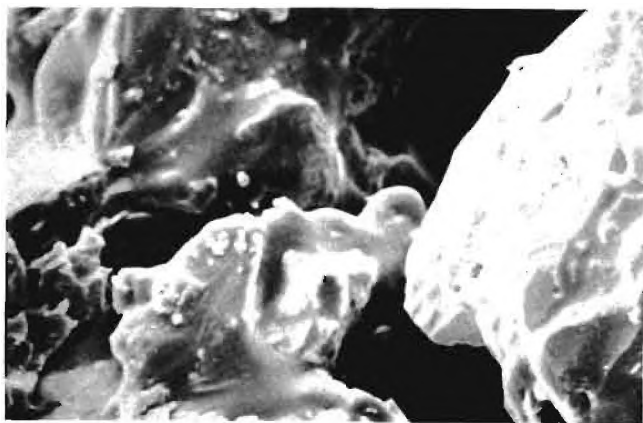


Fig. 8. Scanning electron micrograph of a Sunnyside, Utah, tar sand sample. Bitumen content: 9% by weight. $\sim 400\times$.

cell ranged from 60 to 75% by weight bitumen, on a dry basis. Moisture content was around 40% by weight. The tailings had an unexpectedly low bitumen content of less than 0.5% by weight. The excellent quality of the tailings is well demonstrated by the scanning electron micrograph presented in Fig. 11, which is to be compared with the photograph of the original feed presented in Fig. 6. In these experiments, about 96% of the bitumen contained in the feed material was recovered in the concentrate and about 92% of the sand was rejected in the tailings. Typical results are presented in Table 4.

Identification of Main Variables and Process Optimization:
The Box and Wilson Algorithm: In accordance with the experimentation strategy outlined previously, the Box and Wilson algorithm was applied to the recovery of bitumen from Asphalt Ridge, Utah, tar sands. The objective for optimization was to maximize the quality of the separation being characterized by the coefficient of separation. Maintaining all other digestion and flotation conditions constant, the coefficient of separation was assumed to be a function of only three

Table 4. Example of Hot-Water Separation Results with an Asphalt Ridge Tar Sand Sample

Experimental Conditions:

Digestion:	Feed Source:	Asphalt Ridge
	Wetting Agent:	NaOH
	Temperature:	95°C
	Percent Solids:	70% by weight tar sands
	Digestion Time:	15 min
	NaOH Concentration:	0.58 mole/liter
	Feed Size:	Extruded to $\sim 3/8$ in.
	Agitation:	750 rpm
Flotation:	Cell Design:	Cylindrical
	Flotation Reagents:	None
	Percent Solids:	20% by weight tar sands
	Agitation:	1000 rpm
	Temperature:	15°C
	Air Flowrate:	3000 (cc/min)
	Flotation pH:	~ 10.5

Calculated Mass Balance:

	Wt, % dry	Grade, % Tar	Grade, % Sand	Recovery, % Tar	Recovery, % Sand
Concentrate	19.05	64.82	35.18	96.39	7.69
Tail	80.95	0.57	99.43	3.61	92.31
Feed	100.00	12.81	87.19	100.00	100.00

Coefficient of Separation = 0.8870

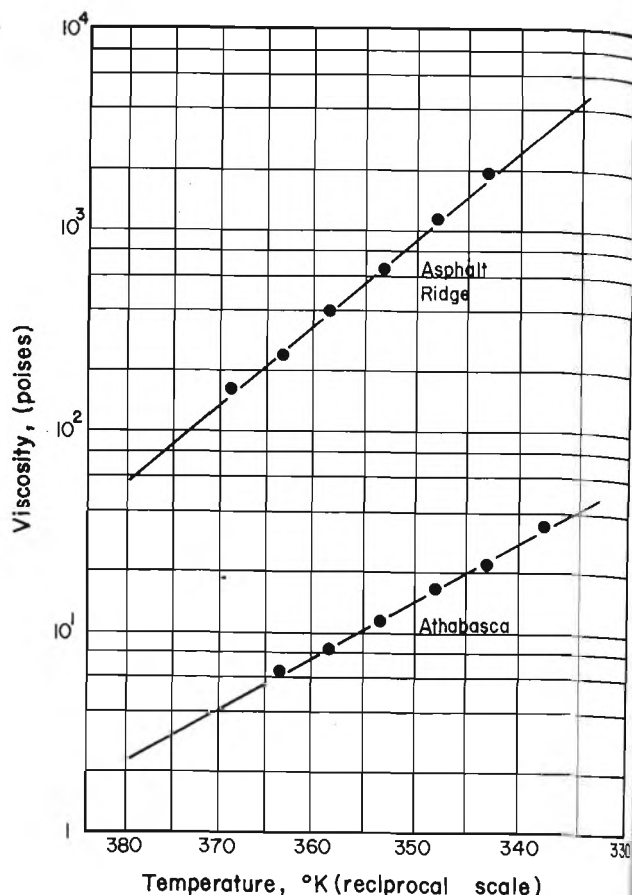


Fig. 9. Arrhenius plot illustrating the effect of temperature on bitumen viscosity.

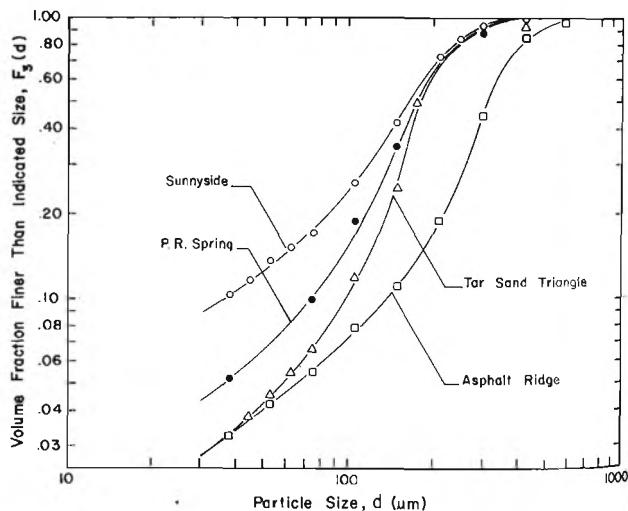


Fig. 10. Particle size distribution of sand from four different Utah tar sand deposits: Tar Sand Triangle, P.R. Spring, Sunnyside, and Asphalt Ridge.

digestion variables; the percent solids in the reactor (V_2), the wetting agent concentration (V_4), and the stirring speed (V_6). Furthermore, based on the Canadian experience with Athabasca tar sands,¹⁵ sodium hydroxide was selected as the wetting agent in all of the experiments. The experimental results obtained during the implementation of the Box and Wilson algorithm are summarized next. Detailed information on the subject may be found elsewhere.²⁹

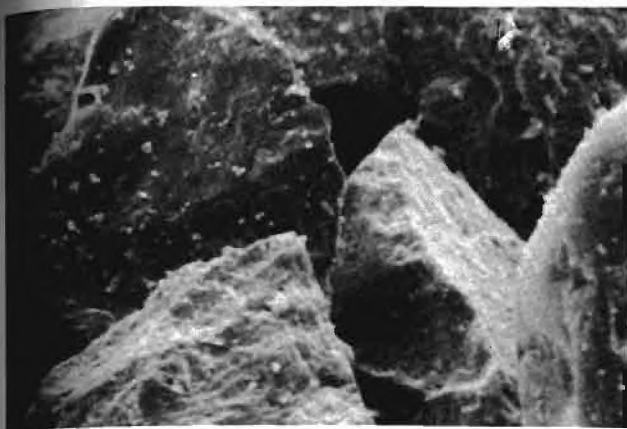


Fig. 11. Scanning electron micrograph of Asphalt Ridge sand after hot-water extraction of the bitumen. ~ 400X.

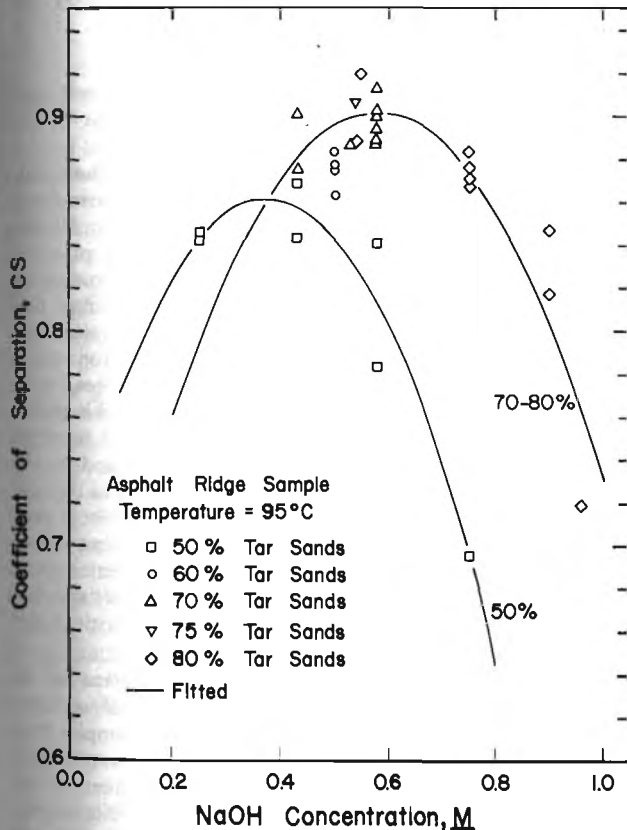


Fig. 12. Quality of the separation achieved by hot-water processing as a function of the caustic concentration, for various percent tar sands in the reactor at 95°C. Asphalt Ridge sample.

The implementation of the Box and Wilson optimization algorithm required more than 60 extraction tests. Percent solids was varied from 50 to 80% by weight, NaOH concentration from 0.25 to 1.50 (mole/liter), and the stirring speed from 510 to 1250 rpm. The experimental results so obtained were correlated by multiple linear regression to obtain the second order experimental model:²⁹

$$CS = 0.9192 - 9.515 \times 10^{-4} V_2 + 1.641 \times 10^{-1} V_4 - 1.694 \times 10^{-4} V_6 + 2.048 \times 10^{-2} V_2 V_4 + 3.825 \times 10^{-6} V_2 V_6 - 1.824 \times 10^{-4} V_4 V_6 - 8.667 \times 10^{-5} V_2^2 - 1.350 V_4^2 - 3.250 \times 10^{-9} V_6^2 \quad (1)$$

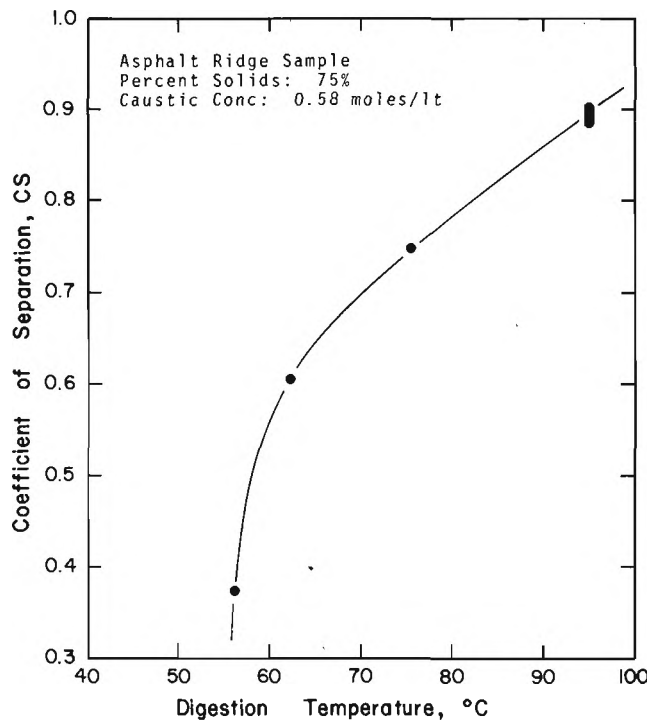


Fig. 13. Quality of the separation as a function of the digestion temperature for the Asphalt Ridge sample at 75% solids and 0.58 mole/liter sodium hydroxide.

where CS represents coefficient of separation (fitted value); V_2 represents percent tar sands, by weight; V_4 represents NaOH concentration in mole/liter; and V_6 represents stirring speed in rpm.

Hypothesis tests, based on the analysis of variance of this model, suggested that, in the range of values studied, the quality of the separation does not seem to be significantly affected by the stirring speed (V_6). In fact, it can be demonstrated²⁹ that the "reduced" model:

$$CS = 0.6553 + 5.796 \times 10^{-3} V_2 - 1.369 \times 10^{-2} V_4 + 1.802 \times 10^{-2} V_2 V_4 - 1.008 \times 10^{-4} V_2^2 - 1.182 V_4^2 \quad (2)$$

which does not account for variations in V_6 , describes the experimental data as well as the "full" model (Eq. 1).

Further analysis of the data reveals that, in the range of 70 to 80% tar sands, the overall system response, characterized by the coefficient of separation, is not significantly affected by the percent tar sands in the digester, V_2 . In fact, above 70% solids, the polynomial expression:

$$CS = 0.5732 + 1.127 V_4 - 0.9698 V_4^2 \quad (3)$$

can be shown²⁹ to describe the experimental data as well as the reduced model correlation (Eq. 2). The quality of the data description by the reduced models is illustrated in Fig. 12 where experimental and fitted values are graphically compared. As indicated there, the maximum coefficient of separation is expected to be around 0.90 at a sodium hydroxide concentration of 0.58 mole/liter. Grades and recoveries are expected to be similar to those reported in Table 4.

The need for high shear digestion conditions in order to achieve both bitumen displacement and phase disengagement gives rise to a relatively high optimum percent solids in the digester (70-80%). Further, experimental results substantiate the hypothesis that a high intensity of agitation does not necessarily result in strong shearing conditions. In fact, as mentioned previously, process performance was shown to be independent of the stirring speed in the range of values studied (510-1250 rpm).

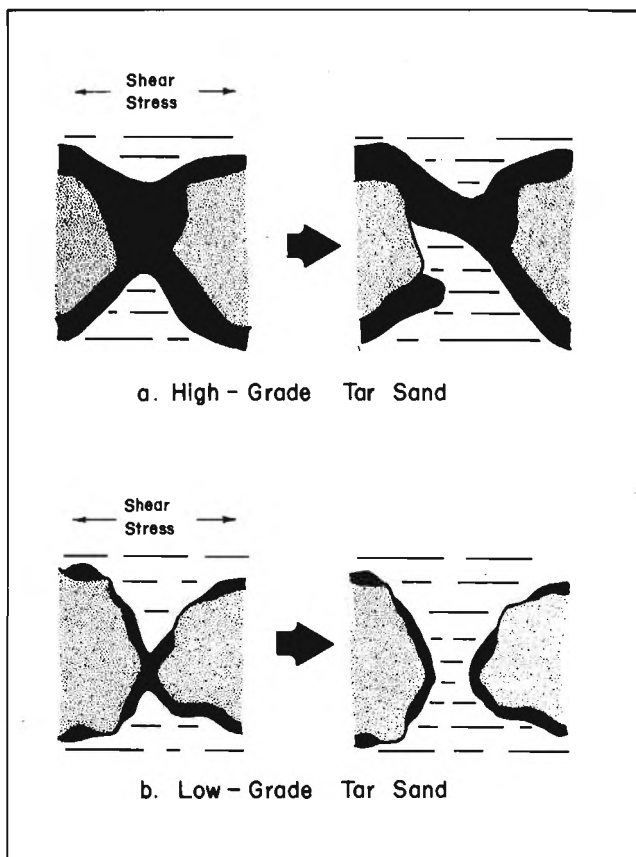


Fig. 14. Schematic representation of the effect of shear forces on the digestion of high and low grade tar sands.

Perhaps the most critical variable is the wetting agent concentration. A low caustic addition (below 0.4M) results in a sticky bitumen concentrate and a tail which still contains a significant amount of bitumen. On the other hand, an excessively high caustic addition (above 1M) gives rise to a strong chemical reaction at the bitumen-water interface. Under these conditions, the tar sand is disintegrated before any phase separation can actually occur.

Effect of Digestion Temperature on the Quality of the Separation: Each of the experiments reported in the previous sections was performed at 95°C. In order to test the hypothesis that the quality of the separation should deteriorate at lower temperatures because of the increase in bitumen viscosity, a series of experiments with Asphalt Ridge tar sand were performed at lower temperatures, setting all other variables at the optimum conditions previously determined. Experimental results presented in Fig. 13 indicate that a decrease in temperature has a detrimental effect on the quality of the separation achieved by hot-water processing. Even though the grade of the concentrate decreased only slightly, the recovery of bitumen from the sand (and hence, the coefficient of separation) was significantly diminished. At these low temperatures, the bitumen became very sticky and viscous. As a result, the bitumen adhered to the walls of the reactor and the flotation cell. Furthermore, large lumps of nonfloatable bitumen-sand agglomerates appeared with the sand tailings.

Effect of the Feed Source on the Quality of the Separation: While excellent results, comparable to those obtained in the processing of Athabasca tar sands, were obtained with the two high-grade Utah samples (Asphalt Ridge and P.R. Spring), hot-water separation tests of low-grade tar sands (Sunnyside and Tar Sand Triangle) were not at all successful. Experimental results obtained with tar sand samples from these four deposits are

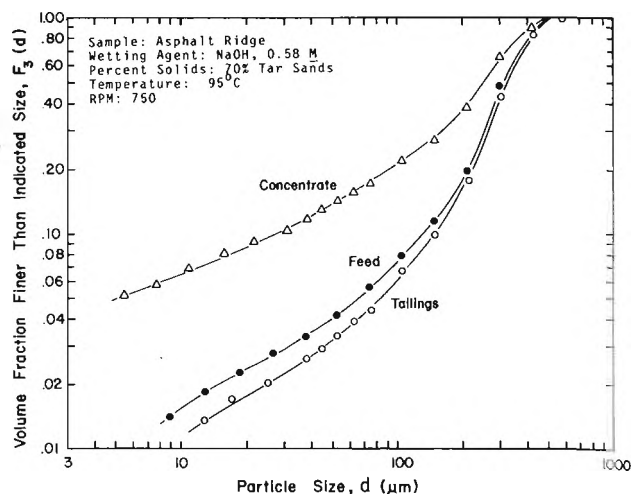


Fig. 15. Particle size distribution of the sand in the feed and products for a typical hot-water extraction test.

summarized in Table 5, where typical results obtained with Athabasca tar sands are also included, as reported in the literature.³⁴

Because of the low bitumen content, samples from Sunnyside and Tar Sand Triangle can be easily ground in a conventional tumbling mill. Such is not the case with samples from Asphalt Ridge and P.R. Spring deposits which due to their plasticity could only be reduced in size to a limited extent by extrusion. As compared to the Sunnyside sample (Fig. 8), the bituminous film in the Athabasca, Asphalt Ridge and P.R. Spring samples (Figs. 5-7, respectively) appears to be much thicker and continuous throughout the sample. With such samples, shear forces can be transferred to the bitumen-solid interface through this plastic bituminous matrix, so that rupture may occur at the interface which will allow for the aqueous solution to advance and wet the surface of the sand particles (Fig. 14a). Conversely, in the case of low-grade tar sands, where the bitumen is more like a thin, rigid coating around each sand particle (Fig. 8) rather than a continuous matrix, failure may occur within the bitumen phase and not at the interface, as postulated in the previous case. As a result, the bitumen remains attached to the sand particles and phase disengagement does not occur (Fig. 14b).

Under the assumption that the bitumen content of the digested pulp was a critical variable, a series of recycling experiments were performed in which tar sand samples from different deposits were mixed with bitumen concentrate prior to hot-water separation, so that the bitumen content of the mixture could be adjusted to any desired level. In this series of experiments, the bitumen concentrate was added directly to the fresh feed in the digester. However, visual observation of the digested pulp revealed that the fresh tar sands and the recycled bitumen concentrate were not mixed thoroughly. In fact, the experimental results presented in Table 5 demonstrate that the quality of the separation was not improved, as anticipated. Alternate techniques, such as heat treatment of the mixture, are currently being investigated by other members of the same research group.

Particle Size Classification during Hot-Water Processing: Particle size analyses of the feed and products after hot-water processing reveals that size classification of the sand particles occurs during the processing sequence. As shown by the size analysis data presented in Fig. 15, most of the fine particles are recovered in the bitumen concentrate whereas coarse particles are rejected to the tailings.

At the optimum digestion conditions, the quality of the separation will be controlled not only by the bitumen content of

Table 5. Effect of the Feed Source on the Quality of the Separation.

Experimental Conditions:

Temperature: 95°C
Percent Solids: 70%
Wetting Agent: 0.58 M, NaOH
Stirring Speed: 750 rpm

Summary of Results:

Source	Bitumen Content, %			Tar	Recovery, %	CS
	Feed	Conc.	Tailings			
Asphalt Ridge	12.8	64.8	0.6	96.4	92.3	0.887
Asphalt Ridge (recycle)	14.6	65.3	0.5	97.1	91.2	0.883
Asphalt Ridge (recycle)	18.3	62.8	0.4	98.3	86.9	0.852
F. R. Spring (*)	14.8	70.5	0.5	97.3	93.0	0.903
Sunnyside	8.9	21.5	1.6	89.0	68.2	0.572
Sunnyside (recycle)	12.7	26.0	1.6	93.3	61.6	0.549
T. S. Triangle	5.0	no products				
T. S. Triangle (recycle)	20.0	no products				
Rebunna (*)	14.3	94.7	1.7	90.0	99.2	0.892

* Wetting agent: 1.75 M sodium silicate
* Experimental conditions not reported.¹⁴

the feed but also by the particle size distribution of the sand in the tar sand sample. For a given bitumen recovery, the influence of these two factors on the quality of the separation of a low-grade Sunnyside sample can be roughly estimated based on the results obtained with the Asphalt Ridge sample under similar conditions (Case 1 in Table 6). Notice that 7.7% of the sand in the Asphalt Ridge sample is recovered in the bitumen concentrate. If the size classification process is assumed to be ideal (i.e., there exists a critical separation size above and below which a given particle reports to either the tailings or the concentrate, respectively), it is found from Fig. 10 or 15 that this critical size must be approximately 100 μ m in order to explain the previously mentioned result. Now, if the same critical size is assumed to be applicable for the Sunnyside sample (Case 2 in Table 6), where 35% of the sand is finer than 100 μ m (Fig. 10), the estimated grades and recoveries shown in Table 6 are found to be in fairly good agreement with the experimental values. Although these highly idealized calculations do not attempt to provide an accurate description of the system, they do demonstrate the overall combined influence of the feed grade and the particle size distribution of the sand on the performance of the hot-water process.

Summary and Conclusions

Experimental results obtained in this investigation demonstrated that effective separation of bitumen from high-grade Utah tar sands can be achieved with a hot-water process, involving the addition of wetting agents, digestion of the tar sand under high shear conditions, and finally separating the bitumen from the sand by a modified froth flotation technique. The processing strategy being developed for Utah tar sands is expected to differ significantly from the one being used in the processing of Athabasca tar sands, mainly because of considerable differences in the physical and chemical properties of the two materials. As compared to Utah tar sands, Athabasca tar sands are characterized by a higher bitumen content, a significantly lower viscosity (about two orders of magnitude lower), and the reported presence of an envelope of connate water separating the bitumen from the solid phase. Utah tar sands contain practically no water; thus, the bitumen is directly in contact with, and bonded to, the surface of the sand particles. Main variables and optimum operating conditions of the

Table 6. Effect of Feed Grade and Particle Size Distribution on the Quality of the Separation (*)

Case 1: Asphalt Ridge (Reference data)
Bitumen Content: 13.1%
"Fines" (>100 μ m): 7.7% (Fig. 10 or 15)

	Weight, %	Grade, %		Recovery, %	
		Tar	Sand	Tar	Sand
Concentrate	19.0	64.8	35.2	96.4	7.7
Tail	81.9	0.6	99.4	3.6	92.3
Feed	100.0	12.8	87.2	100.0	100.0

Case 2: Sunnyside
Bitumen Content: 9.6%
"Fines" (>100 μ m): 25% (Fig. 10)

	Weight, %	Grade, %		Recovery, %	
		Tar	Sand	Tar	Sand
Concentrate	36.9 (30.7)	21.5 (25.7)	78.5 (74.3)	89.0 (89.0)	31.8 (25.0)
Tail	63.1 (69.3)	1.5 (1.4)	98.4 (98.6)	11.0 (11.0)	68.2 (75.0)
Feed	100.0	8.9	91.1	100.0	100.0

* Calculated values in parenthesis.

digestion stage were determined for the Asphalt Ridge sample by the implementation of the Box and Wilson optimization algorithm. The quality of the separation, being characterized by the coefficient of separation, was found to be independent of the stirring speed in the range of values considered (510-1250 rpm). At the optimum digestion conditions (70-80% tar sands, 0.58 M NaOH, 95°C), coefficients of separation averaging 0.90 and bitumen recoveries over 96% are to be expected. Separation test data obtained at lower digestion temperatures substantiated the hypothesis that a decrease in temperature has a detrimental effect on the quality of the separation achieved by hot-water processing.

Variations in the quality of the separation obtained with materials from different deposits can be interpreted in terms of their physical properties, specifically the bitumen content and the particle size distribution of the sand. As discussed in the text, the grade of the tar sand must be high enough so that the bituminous film surrounding the sand particles exceeds a certain critical thickness for effective phase disengagement. In addition, the particle size distribution of the sand must be coarse enough so that the amount of fines entrapped in the bitumen phase as a result of the size classification process is minimized and the grade of the bitumen concentrate is maintained at an acceptable level.

Although preliminary in nature, the experimental evidence gathered in this investigation seems to indicate that a hot-water process for Utah tar sands would allow for the efficient utilization of this important energy resource.

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